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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : B01D 21/01 — A1

(11) International Publication Number: **WO 99/61129**

(43) International Publication Date: 2 December 1999 (02.12.99)

(21) International Application Number: PCT/US99/10961

(22) International Filing Date: 19 May 1999 (19.05.99)

(30) Priority Data:
PP 3704 25 May 1998 (25.05.98) AU

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(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: DEXTRAN STARCH AND FLOCCULANT COMBINATION FOR IMPROVING RED MUD CLARIFICATION

(57) Abstract

The claimed invention is a method for separating Bayer process red mud from a Bayer process liquor which comprises adding to a Bayer process liquor containing red mud an effective amount of a water soluble synthetic flocculant, dextran and starch combination. The flocculant is added anywhere in the slurry containing the red mud suspended in Bayer process liquor, or in a liquor slurry containing bauxite prior to or during digestion. Once the flocculant combination is added, it is mixed sequentially with the Bayer process liquor and the red mud contained in the Bayer process liquor is removed by sedimentation, centrifugation or filtration.

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DEXTRAN STARCH AND FLOCCULANT COMBINATION FOR IMPROVING RED MUD CLARIFICATION

In the Bayer process for the production of alumina, bauxite ore is pulverized, slurred in water, and then digested with caustic at elevated temperatures and pressures. The caustic solution dissolves oxides of aluminum, forming an aqueous sodium aluminate solution. The caustic-insoluble constituents of bauxite ore (referred to as red mud") are then separated from the aqueous phase containing the dissolved sodium aluminate. Solid alumina trihydrate product is precipitated out of the solution and collected as product.

In more detail, the pulverized bauxite ore is fed to a slurry mixer where a water slurry is prepared. The slurry makeup water is typically spent liquor (described below) and added caustic. This bauxite ore slurry is then diluted and passed through a digester or a series of digesters where alumina is released from the ore as caustic-soluble sodium aluminate. The digested slurry is then cooled to about 110°C (about 230°F), typically employing a series of flash tanks wherein heat and condensate are recovered. The aluminate liquor leaving the flashing operation contains from about 1 to about 20 weight percent suspended solids, which solids consist of the insoluble residue that remains after, or is precipitated during, digestion. The coarser solid particles may be removed from the aluminate liquor with a "sand trap" cyclone. The finer solid particles are generally separated from the liquor first by settling and then by filtration, if necessary. The slurry of aluminate liquor and the finer solids is normally first fed to the center well of a mud settler, or primary settler, where it is treated with a flocculant, and as the mud settles, clarified sodium aluminate solution,

referred to as "green" or "pregnant" liquor, overflows a weir at the top. This overflow from the mud settling tank is passed to the subsequent process steps. If the aluminate liquor overflowing the settler contains an unacceptable concentration of suspended solids (at times from about 50 to about 500 mg of suspended solids per liter), it is then generally further clarified by filtration to give a filtrate with no more than about 10 mg suspended solids per liter of liquor. The treatment of the liquor collected after the primary settlement to remove any residual suspended solids before alumina trihydrate is recovered is referred to as a secondary clarification stage.

The clarified sodium aluminate liquor is seeded with alumina trihydrate crystals to induce precipitation of alumina in the form of alumina trihydrate, $AL(OH)_3$. The alumina trihydrate particles or crystals are then separated from the concentrated caustic liquor, and the remaining liquid phase, the spent liquor, is returned to the initial digestion step and employed as a digestant after reconstitution with caustic.

In another section of the Bayer circuit, the settled solids of the primary settler ("red mud") are withdrawn from the bottom of the settler and passed through a countercurrent washing circuit for recovery of sodium aluminate and soda. The countercurrent washing circuit utilizes two or more washers which receive a mud washer feed slurry from either the settler underflow or other washer underflow, as well as any dilution liquor. As noted above, the red mud does not include any coarser particles removed prior to feeding the slurry to the primary or mud settler.

The at least partial separation of the red mud solids from the pregnant liquor at elevated temperatures

by settling or by filtration is expedited by the use of a flocculant. This initial clarification of the pregnant liquor into a clarified liquor phase is referred to as the primary settler state. Flocculating agents improve the separation of insolubles by increasing the rate at which the solids settle, by reducing the amount of residual solids suspended in the liquor, and by decreasing the amount of the liquor in the settled solids phase. Flocculation performance is highly important in the primary settlement stages. Red muds are comprised chiefly of iron oxides (at least about 50 weight percent of the red mud solids), together with silicon oxides, calcium oxides, sodium alumino-silicates, titanium oxides and other materials, and commonly represent from about 5 to about 50 percent of the dry weight of the bauxite ore. Generally these muds are comprised of very fine particles, which hinder the desired rapid and clean separation of red mud particles from the solublized alumina liquor. If the rate of separation is slow, output is materially diminished and overall process efficiency is impaired. If the separation is not clean, the resultant aluminate liquor will require a more extensive treatment to remove residual solids, and/or the alumina trihydrate recovered will contain levels of impurities that are undesirably high for many end uses.

The polysaccharides starch and dextran have, for some time, been used in red mud flocculation. For instance, U.S. Patent No. 3,085,853, April 16, 1963, Lesinski et al., uses native dextrans to increase the rate of sedimentation of finely divided solids in aqueous suspensions and thereby facilitate the separation of such solids. Later synthetic polymeric flocculants became more commonly employed for the Bayer process. U.S. Patent No. 3,390,959 issued July 2, 1968 to Sibert, uses

acrylate homopolymers and copolymers which contain not more than 20% of other ethylenically unsaturated polymerizable polar monomers for the Bayer process. Included in Siber's polar commoners are acrylamide and diethylvinylphosphonate, among others. Diethylvinylphosphonate is the diethyl ester of vinylphosphonic acid, and can be hydrolyzed to the monoethyl ester in caustic solution.

U.S. Patent No. 3,397,953, August 20, 1968, Galvin et al., uses a blend of starch and polyacrylic acid on red mud suspensions, noting that polyacrylic acid alone is not suitable as a flocculating agent. The polyacrylic acids exemplified generally have molecular weights of less than 300,000. The flocculation and sedimentation activity of the blend is exemplified in the primary settler stage of a bauxite process. U.S. Patent No. 3,445,187, May 20, 1969, Sibert, uses synthetic acrylic acid polymer alone to enhance the rate of separation of red mud solids from the aqueous caustic solutions during secondary clarification steps. The synthetic polymer used contains at least about 80 weight percent of the acrylic acid mer unit, and has a molecular weight in excess of 50,000, and preferably in excess of 100,000. U.S. Patent No., 3,541,009, November 17, 1970, Arendt et al., uses a combination of causticized or modified starch, a water soluble polymer, and a caustic alkali to enhance the coagulation, sedimentation and/or filtration of aqueous suspensions of solids, including the settling of red mud from Bayer process liquor. The water soluble polymer is derived from at least one olefinically-unsaturated monomer and has a molecular weight in excess of 100,000.

U.S. Patent No. 3,681,012, August 1, 1972, Sibert, uses acid acrylic polymer most preferably having

molecular weight of at least, 1,000,000, either alone or in combination with starch, for clarification of digested bauxite containing solublized alumina and red mud residues. U.S. Patent No. 4,767,540, August 30, 1988, Spitzer et al., uses a polymer that contains hydroxamic acid groups for the same purpose. U.S. Patent No. 5,008,089, April 16, 1991, Moody et al., uses a combination of dextran and synthetic anionic polymer for flocculating red mud in Bayer process liquors.

U.S. Patent No. 5,217,620, June 8, 1993, Mahoney et al., uses a combination of pullulan, lacatan, rhamsan, or zooglan with a conventional water soluble anionic flocculant for red mud settling.

The synthetic flocculating agents employed for the settling of filtration of red mud are generally water soluble polymers of one or more ethylenically-unsaturated monomers, and have been used together, as noted above, with starch or dextran for aluminate liquor clarification. The synthetic flocculating agents are usually anionic, and the optimum anionic content of such polymer is usually related to the alkalinity of the liquor. In the washing circuit, the early wash liquors have the highest alkalinity and may require a more highly anionic polymer than the later wash liquors.

It is an object of the present invention to provide a more effective flocculation for separating red mud from the red mud-containing liquors, particularly preferably the primary settler liquor, of the Bayer process. It is a preferred object of the present invention to provide an improved method whereby the suspended solids retained in the supernatant phase after flocculation of the red mud-containing liquors, particularly the primary settler liquor, of the Bayer process are diminished. It is further preferred objects of the present invention to

provide a more effective Bayer process wherein flocculation for separating red mud from the red mud-containing liquors particularly the primary settler liquor, is improved by a more complete flocculation of the solids.

DISCLOSURE OF THE INVENTION

In a first aspect, the present invention provides a method for treating Bayer process liquor containing red mud comprising contacting the Bayer process liquor with, in combination, an effective amount of a water soluble synthetic flocculant, dextran and starch prior to separating the red mud from the liquor.

In a second aspect, the present invention provides an agent for treatment of Bayer process liquor containing red mud said agent comprising, in combination, a water soluble synthetic flocculant, dextran and starch in amounts effective to increase separation of the red mud from the Bayer process liquor.

The combination preferably contacts the slurry containing the red mud suspended in Bayer process liquor, or a liquor slurry containing bauxite prior to or during digestion. The dextran, starch and flocculant combination can be added to the Bayer process liquor separately or together provided that in at least one point of the process a combination of all three components are present in the Bayer process liquor. If the three components are added separately, they may be added in any order, but it is preferred to add the starch and polymer (separately or together) prior to the addition of the dextran. In preferred embodiments, the starch and polymer are added to the process upstream from the point of addition of the dextran.

Once the components of the combination are added, they are mixed sequentially with the Bayer process liquor, and the red mud contained in the Bayer process liquor is removed by sedimentation, centrifugation or filtration.

Water-soluble synthetic flocculants which may be used in combination with dextran and starch include, but are not limited to acrylates, homopolymers of acrylic acid, copolymers of acrylic acid and acrylamide and copolymers of acrylic acid and acrylamide modified to contain a hydroxamic acid or acrylic acid moieties. Particularly preferred are ammonium acrylate polymers because of their replacement ratio and apparent synergy. The red mud thus treated may be separated from the liquor phase using a separator selected from the group consisting of settlers, thickeners, centrifuges and filters.

Preferably, the combination which contacts the Bayer process liquor is used in an amount of from about 0.01 to about 10 grams per liter of Bayer process liquor treated. The combination is more preferably used in an amount of from about 0.1 to about 2 grams per liter of liquor treated. The combination may contact the Bayer process liquor anywhere. For example the combination may contact the Bayer process liquor at a point selected from the group consisting of the primary settler feed, bauxite pretreatment, bauxite digestion and flash tanks. As stated above the dextran, starch and polymer may be added to the liquor separately or together. Preferably the starch and polymer may be added to the liquor separately as far back upstream from the addition of dextran as possible (the further back, the better for clarity reduction). For example the starch and polymer can be added as one solution or separately to a thickener feed

line followed by addition of dextran to the feed line just prior to the feedwell or into the feedwell via sparges. Preferably the combination contacts the Bayer Process liquor in the primary settler feed.

BRIEF DESCRIPTION OF DRAWINGS

The present invention will now be described by way of reference to the following non-limitative examples and drawings in which:

Figure 1 is a graph comparing dosage of starch in grams per tonne (GPT) to reduction in clarity and

Figure 2 is a graph comparing additions of various constituents in grams per tonne and their effect on clarity.

BEST MODE FOR CARRYING OUT THE INVENTION

To evaluate the effectiveness of the combination, settling tests were completed in a waterbath with temperature maintained at 96°C. Eighteen (18) cylinder of identical mud/liquor characteristics were tested during one experiment.

High Molecular Weight Homopolmyer Ammonium Acrylate (hereinafter referred to as Polymer A) with a molecular weight greater than 10 million and supplied by Nalco was diluted in spent liquor to a concentration of 1.5 gpl (0.15%) by introducing the neat polymer to the vortex produced by a cage stirrer at 800 RPM and mixing four five (5) minutes. Dextran (hereinafter referred to as Polymer B) was diluted in lake water to a concentration of either 5 or 10 gpl (0.5 or 1.0%) by gentle mixing by shaking the bottle by hand. Starch was supplied as a 400 gpl (40%) solution and diluted with lakewater to 100 gpl (10%), and then further diluted with spent liquor to a final concentration of 20 gpl (2%) again by shaking the bottle by hand.

Polymer and Starch solutions were added together and the cylinders mixed using a gang plunger which allows six (6) cylinders to be tested at one time. After addition and mixing of polymer and starch solutions the dextran solution was added and further mixing completed using the gang plunger.

Settling rate is presented in m/h and determined by measuring the time for flocculated mud particles to settle from 1000 ml to 600 ml in a 1000 ml cylinder.

Clarity was determined by decanting 250 ml of supernatant from the top of the settled cylinder after 30 minutes, adding 75 ml of 10N NaOH and boiling mixture to negate any precipitation of hydrate. After boiling, the liquor was cooled and passed through a turbidity meter and clarity determined and presented in NTU units.

The results of these tests showing the various synthetic flocculant/starch/dextran dosages are shown in tables 1 and 2.

TABLE 1

Cylinder	Plunges After Polymer/ Starch Addition	Plunges After Dextran Addition	Polymer Dose (ml)	Polymer Dose (gpt)	Starch Dose (ml)	Starch Dose (gpt)	Dextran Dose (ml)	Dextran Dose (gpt)	Settling Rate (m/h)	Clarity (NTU)	Reduction in Clarity (%)
1	20	10	4.0	150	0.0	0	0.4	100	2.9	220	0
2	20	10	4.0	150	0.5	250	0.4	100	3.4	215	2
3	20	10	4.0	150	1.0	500	0.4	100	4.1	210	5
4	20	10	4.0	150	1.5	750	0.4	100	4.1	200	9
5	20	10	4.0	150	2.0	1000	0.4	100	4.5	200	9
6	20	10	4.0	150	3.0	1500	0.4	100	4.9	155	30
7	20	10	4.0	150	0.0	0	0.8	200	3.8	245	0
8	20	10	4.0	150	0.5	250	0.8	200	4.4	225	8
9	20	10	4.0	150	1.0	500	0.8	200	4.4	215	12
10	20	10	4.0	150	1.5	750	0.8	200	4.5	210	14
11	20	10	4.0	150	2.0	1000	0.8	200	4.5	185	24
12	20	10	4.0	150	3.0	1500	0.8	200	5.1	145	41
13	20	10	4.0	150	0.0	0	1.6	400	4.5	215	0
14	20	10	4.0	150	0.5	250	1.6	400	4.6	205	5
15	20	10	4.0	150	1.0	500	1.6	400	5.4	160	26
16	20	10	4.0	150	1.5	750	1.6	400	5.6	140	35
17	20	10	4.0	150	2.0	1000	1.6	400	5.8	115	47
18	20	10	4.0	150	3.0	1500	1.6	400	6	95	56

TABLE 2

Cylinder	Plunges After Polymer/ Starch Addition	Plunges After Dextran Addition	Polymer Dose (ml)	Polymer Dose (gpt)	Starch Dose (ml)	Starch Dose (gpt)	Dextran Dose (ml)	Dextran Dose (gpt)	Settling Rate (m/h)	Clarity (NTU)	Reduction in Clarity (%)
1	30	10	6.0	225	0.0	0	0	0	1.7	235	0
2	30	10	6.0	225	0.5	250	0	0	2.2	185	21
3	30	10	6.0	225	1.0	500	0	0	3.2	200	15
4	30	10	6.0	225	2.0	1000	0	0	3.6	215	9
5	30	10	6.0	225	3.0	1500	0	0	4.6	190	19
6	30	10	6.0	225	4.0	2000	0	0	4.6	160	32
7	30	10	6.0	225	0.0	0	1	250	3	210	0
8	30	10	6.0	225	0.5	250	1	250	3.7	200	5
9	30	10	6.0	225	1.0	500	1	250	4.5	200	5
10	30	10	6.0	225	2.0	1000	1	250	4.6	140	33
11	30	10	6.0	225	3.0	1500	1	250	6.4	110	48
12	30	10	6.0	225	4.0	2000	1	250	4.6	90	57
13	30	10	6.0	225	0.0	0	2	500	4.1	230	0
14	30	10	6.0	225	0.5	250	2	500	4.1	195	15
15	30	10	6.0	225	1.0	500	2	500	4.8	165	28
16	30	10	6.0	225	2.0	1000	2	500	4.5	135	41
17	30	10	6.0	225	3.0	1500	2	500	5.4	65	72
18	30	10	6.0	225	4.0	2000	2	500	5	50	78

The results are also shown in the attached Figure 1. It can be seen from the figure that including dextran and polymer in a starch flocculant combination resulted in a marked improvement in clarity.

To highlight the synergistic effect of the various constituents of the treatment agent, further tests were carried out with various dosages of the water soluble synthetic flocculant, starch and dextran. The synthetic flocculant (hereinafter referred to as Nalco 85111) was a high molecular weight ammonium acrylate with a molecular weight greater than 10 million made up at 0.18% solution in lake water. The starch was made up to 1.1% in spent liquor as per conventional plant practice. The dextran (hereinafter referred to as Nalco 85711) had a molecular weight of greater than 500,000 and was made up as a 1% solution in lake water.

As with the previous examples, the polymer and starch were added first. The combination of slurry, starch and Nalco 85111 were mixed by plunging 10 times and then the Nalco 85711 dextran added and mixed by plunging a further five times.

The clarity tests were conducted in a manner similar to the aforementioned examples, however, the clarity was determined after five minutes to allow the differences in dosages to be more clearly identified.

Results of the tests are shown in Table 3 and figure 2.

TABLE 3 - COMPARATIVE EXAMPLES

Eg	85111 Conc (gpl)	85111 Dose (ml)	Starch Conc (gpl)	Starch Dose (ml)	85711 Conc (gpl)	85711 Dose (ml)	Feed Solids (gpl)	Floc Dose (gpt)	Starch Dose (gpt)	85711 Dose (gpt)	Srate T600 (secs)	Srate (m/h)	O/F Clarity NTU
1	1.8	1.5	11	9	1	20	40	68	990	500	45	11.1	80
2	1.8	0	11	9	1	20	40	0	990	500	300	1.7	185
3	1.8	1.5	11	9	1	0	40	68	990	0	57	8.8	275
4	1.8	1.5	11	0	1	20	40	68	0	500	64	7.8	135
5	1.8	0	11	9	1	0	40	0	990	0	600	0.8	250
6	1.8	0	11	0	1	20	40	0	0	500	-	<0.2	1000
7	1.8	1.5	11	0	1	0	40	68	0	0	80	6.3	370

The synergistic effect of the three constituent making up the treatment agent will be clear from these results and in particular figure 2. As can be seen from this figure, in each example where one of the constituent is left out, there is a substantial reduction in the clarity determined after five minutes. The closest comparative example is Example 4 in which only dextran and the water soluble synthetic flocculant are added to the Bayer process liquor. In this instance, clarity is measured at 135 NTU. This is nearly 70% higher than Example 1 using the proposed treatment agent (clarity 80 NTU). Other comparative examples are between two and 12 times less effective.

It will be clear to persons skilled in the art therefore that the combination of water soluble synthetic flocculant, dextran and starch provides a significant increase in the effectiveness of separation processes particularly sedimentation, centrifugation and filtration which is unrecognized and hitherto unsuspected from the prior art.

This invention also relates to the use of the combination of water soluble synthetic flocculant, dextran and starch for promoting coagulation or flocculation in other mineral slurries such as coal, kaolin, copper, precious metals, phosphate, taconite and refuse tailings obtained from these ores.

Changes can be made in the composition, operation and arrangement of the method of the present invention described herein without departing from the concept and scope of the invention as defined in the following claims.

CLAIMS

1. A method for treating Bayer process liquor containing red mud comprising contacting the Bayer process liquor with, in combination, an effective amount of a water soluble synthetic flocculate, dextran and starch prior to separating the red mud from the liquor.

2. A method according to claim 1 wherein the red mud is separated from the liquor by a process selected from the group consisting of sedimentation, centrifugation and filtration.

3. A method according to claim 1 wherein the water soluble synthetic flocculant, dextran and starch combination is used in an amount of from about 0.01 to about 10 grams per liter of liquor treated.

4. A method according to claim 1 wherein the water soluble synthetic flocculant, dextran and starch combination is used in an amount of from about 0.1 to about 2 grams per liter of liquor treated.

5. A method according to claim 1 wherein the water soluble synthetic flocculant, dextran or starch are added separately or together to the Bayer process liquor.

6. A method according to claim 1 wherein the water soluble synthetic flocculant and starch are added together to the Bayer process liquor and one solution and separate from the dextran.

7. A method according to claim 1 wherein the water soluble synthetic flocculant and starch are added together to the Bayer process liquor upstream of the dextran addition to the Bayer process liquor.

8. A method according to claim 1 wherein the water soluble synthetic flocculant, dextran and starch

combination contacts the Bayer process liquor at one or more points selected from the group consisting of primary settler feed, bauxite pretreatment, bauxite digestion and the flask tanks.

9. A method according to claim 1 wherein the step of separating the red mud from the liquor is carried out by a separator selected from the group consisting of settlers, thickeners, centrifuges and filters.

10. A method according to claim 1 wherein the water soluble synthetic flocculant is selected from the group consisting of homopolymers of acrylic acid, copolymers of acrylic acid and acrylamide, copolymers of acrylic acid and acrylamide modified to contain a hydroxamic acid moiety and copolymers of acrylic acid and acrylamide modified to contain an acrylic acid moiety.

11. An agent for treatment of Bayer process liquor containing red mud said agent comprising, in combination, a water soluble synthetic flocculant, dextran and starch in a quantity sufficient to increase separation of the red mud from the Bayer process liquor.

12. An agent as claimed in claim 11 wherein the water soluble synthetic flocculant, dextran and starch combination is used in an amount of from about 0.01 to about 10 g/l of liquor treated.

13. An agent as claimed in claim 11 wherein the water soluble synthetic flocculant, dextran and starch combinations is used in an amount of from 0.1 to about 2 g/l of liquor treated.

14. An agent as claimed in claims 11 wherein the agent comprises two components, a first component comprising water soluble synthetic flocculant and starch and a second component comprising dextran, the two components being suitable for separate addition to the Bayer process liquor.

15. An agent as claimed in claim 11 wherein the water soluble synthetic flocculant is selected from the group consisting of homopolymers of acrylic acid, copolymers of acrylic acid and acrylamide, copolymers of acrylic acid and acrylamide modified to contain hydroxamic acid moiety and copolymers of acrylic acid and acrylamide modified to contain an acrylic acid moiety.

FIG. 1

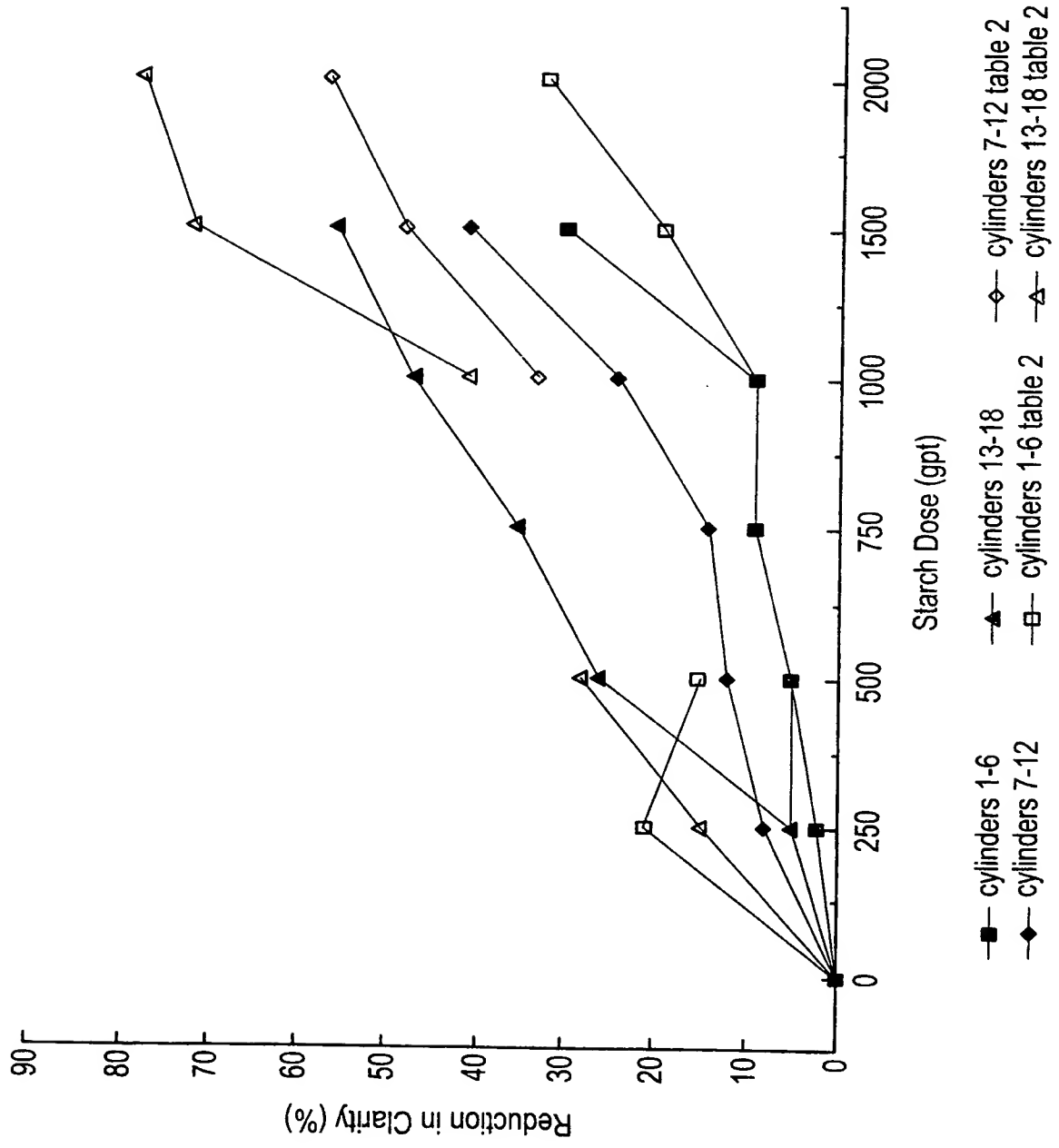
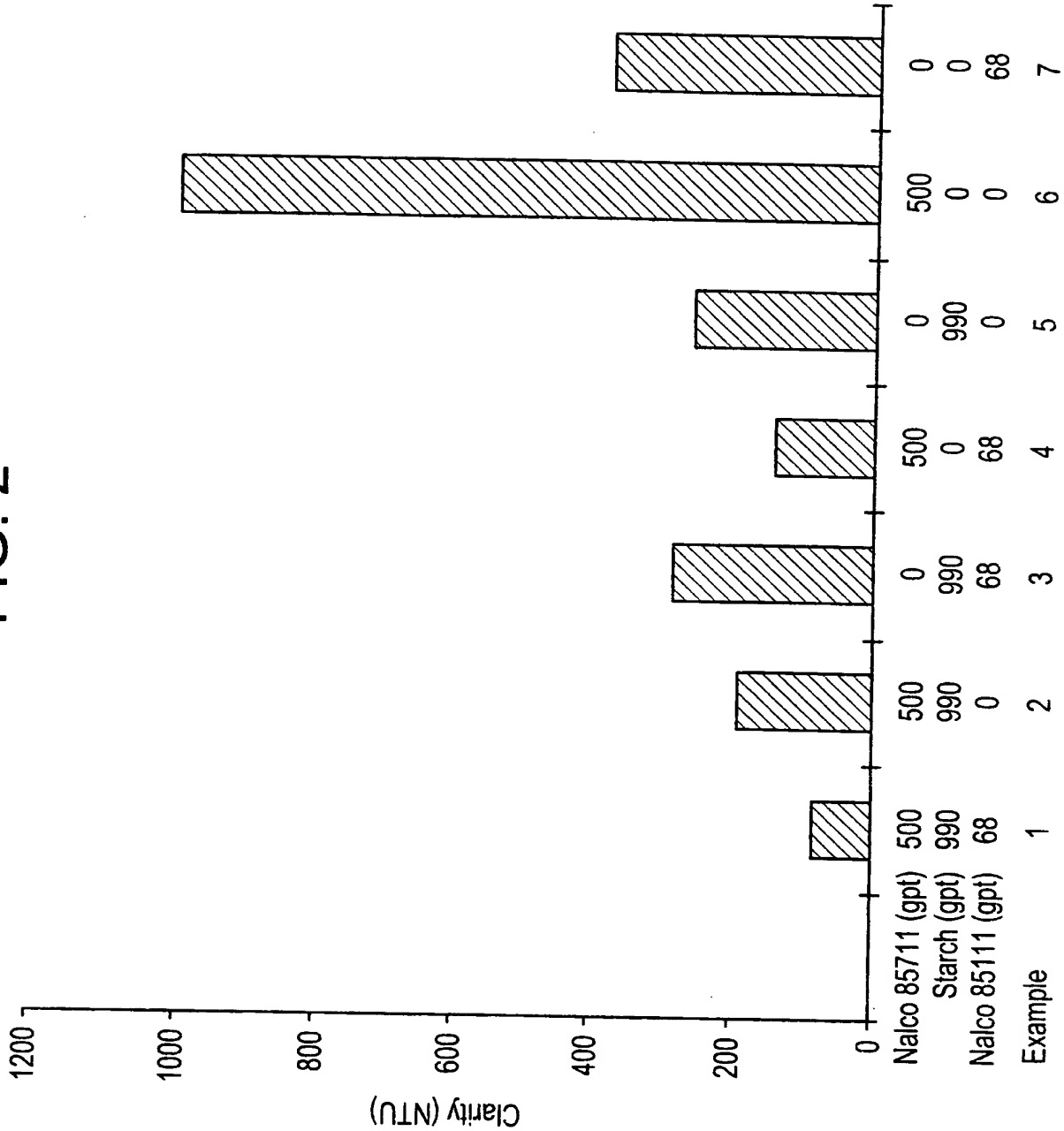

Reduction in Clarity (%)
0
10
20
30
40
50
60
70
80
90
0
250
500
750
1000
1500
2000
Starch Dose (gpt)
cylinders 1-6
cylinders 7-12
cylinders 13-18
cylinders 1-6 table 2
cylinders 7-12 table 2
cylinders 13-18 table 2

FIG. 2


Clarity (NTU)
1200
1000
800
600
400
200
0
Nalco 85711 (gpt)
Starch (gpt)
Nalco 85111 (gpt)
Example
500 990 68 1
500 990 0 2
0 990 68 3
500 0 68 4
0 990 0 5
500 0 0 6
0 0 68 7

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/10961

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :B01D 21/01
US CL :210/728

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 210/728, 730, 731, 732, 733, 734; 252/60, 180, 181; 423/121, 122, 130, 131; 536/102,112

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

NONE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,516,435 A (LEWELLYN) 14 May1996, col. 3 lines 25-67, col. 10 lines 15-29.	1-15
Y		1-15
A	US 5,008,089 A (MOODY et al.) 16 April 1991.	
A	US 3,541,009 A (ARENDT et al.) 17 November 1970.	

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search 06 AUGUST 1999	Date of mailing of the international search report 31 AUG 1999
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer PETER A. HRUSKOCI Telephone No. (703) 308-3839

Form PCT/ISA/210 (second sheet)(July 1992)★

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REC'D 24 JUL 2000
WIPO PCT

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference AU3704	**FOR FURTHER ACTION**	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/US99/10961	International filing date *(day/month/year)* 19 MAY 1999	Priority date *(day/month/year)* 25 MAY 1998
International Patent Classification (IPC) or national classification and IPC IPC(7): B01D 21/01 and US Cl.: 210/728		
Applicant NALCO CHEMICAL COMPANY		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets.

   ☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority. (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 0 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of report with regard to novelty, inventive step or industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand 14 OCTOBER 1999	Date of completion of this report 24 MAY 2000
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer PETER A. HRUSKOCI DEBORAH THOMAS PARALEGAL SPECIALIST Telephone No. (703) 308-0661

Form PCT/IPEA/409 (cover sheet) (July 1998)★

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US99/10961

I. Basis of the report

1. With regard to the elements of the international application:*

[X] the international application as originally filed

[X] the description:
pages 1-14, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____

[X] the claims:
pages 15-17, as originally filed
pages NONE, as amended (together with any statement) under Article 19
pages NONE, filed with the demand
pages NONE, filed with the letter of _____

[X] the drawings:
pages 1-2, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____

[X] the sequence listing part of the description:
pages NONE, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:

[ ] the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

[ ] the language of publication of the international application (under Rule 48.3(b)).

[ ] the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

[ ] contained in the international application in printed form.

[ ] filed together with the international application in computer readable form.

[ ] furnished subsequently to this Authority in written form.

[ ] furnished subsequently to this Authority in computer readable form.

[ ] The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

[ ] The statement that the information recorded in computer readable form is identical to the writen sequence listing has been furnished.

4. [X] The amendments have resulted in the cancellation of:

[X] the description, pages NONE

[X] the claims, Nos. NONE

[X] the drawings, sheets/~~fig~~ NONE

5. [X] This report has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* *Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

** *Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.*

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US99/10961

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. statement

Novelty (N)	Claims	4, 6, 7	YES
	Claims	1-3, 5, 8-15	NO
Inventive Step (IS)	Claims	NONE	YES
	Claims	1-15	NO
Industrial Applicability (IA)	Claims	1-15	YES
	Claims	NONE	NO

2. citations and explanations (Rule 70.7)

Claims 1-3, 5, and 8-15 lack novelty under PCT Article 33(2) as being anticipated by Lewellyn. It is submitted that Lewellyn disclose (see col. 3 lines 25-67 and col. 10 lines 15-45) a method and agent for treating Bayer process liquor as recited in the instant claims.

Claims 4, 6, and 7 lack an inventive step under PCT Article 33(3) as being obvious over Lewellyn. The claims differ from Lewellyn by reciting the use of a specific amount of the combination, and the addition of the flocculant and starch together as a solution separate from the dextran, and together upstream of the dextran addition. It is well known in the art of liquid purification to regulate the amount of flocculant used and the sequence of addition, based on the solids content and the electrical charge of the solids in the liquid being treated, respectively. It would have been an obvious matter of process optimization to one skilled in the art of liquid purification to modify the method of Lewellyn by adding the recited amount and by utilizing the separate and sequential addition, depending on the specific process liquor treated and results desired, absent a sufficient showing of unexpected results.

Claims 4, 6, and 7 meet the criteria set out in PCT Article 33(2), because the prior art does not disclose a method for treating Bayer process liquor, using the specific amount of the combination, or adding the flocculant and starch together as a solution separate from the dextran, or together upstream of the dextran addition, as recited in the instant claims.

Claims 1-15 have industrial applicability and meet the criteria set out in PCT Article 33(4), because the subject matter as claimed can be made or used in industry to treat Bayer process liquors.

---------- NEW CITATIONS ----------

NONE

International application No.
PCT/US99/10961

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

Claim 1 is objected to under PCT Rule 66.2(a)(v) as lacking clarity under PCT Article 6 because the claim is indefinite for the following reason(s): In claim 1 "flocculate" is erroneous and should be changed to - flocculant -.

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

I. BASIS OF REPORT:

5. (Some) amendments are considered to go beyond the disclosure as filed:
NONE

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

gp

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Assistant Commissioner for Patents
United States Patent and Trademark Office
Box PCT
Washington, D.C.20231
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

Date of mailing: 02 December 1999 (02.12.99)	
International application No.: PCT/US99/10961	Applicant's or agent's file reference: 5436
International filing date: 19 May 1999 (19.05.99)	Priority date: 25 May 1998 (25.05.98)
Applicant: BARHAM, Scott et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International preliminary Examining Authority on:

14 October 1999 (14.10.99)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer: J. Zahra Telephone No.: (41-22) 338.83.38

Copy for the Elected Office (EO/US)

PCT/US99/10961

PATENT COOPERATION TREATY

PCT

From the INTERNATIONAL BUREAU

To:

KEEFER, Timothy, J.
Wildman, Harrold, Allen & Dixon
225 West Wacker Drive
Chicago, IL 60604-1229
ETATS-UNIS D'AMERIQUE

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

Date of mailing (day/month/year)
14 July 2000 (14.07.00)

Applicant's or agent's file reference
5436

IMPORTANT NOTIFICATION

International application No.
PCT/US99/10961

International filing date (day/month/year)
19 May 1999 (19.05.99)

1. The following indications appeared on record concerning:

☐ the applicant ☐ the inventor ☒ the agent ☐ the common representative

Name and Address
KEEFER, Timothy, J.
Ladas & Parry
224 S. Michigan Avenue
Chicago, IL 60604
United States of America

State of Nationality

State of Residence

Telephone No.
(312) 427-1300

Facsimile No.
(312) 427-6663

Teleprinter No.
203649

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

☐ the person ☐ the name ☒ the address ☐ the nationality ☐ the residence

Name and Address
KEEFER, Timothy, J.
Wildman, Harrold, Allen & Dixon
225 West Wacker Drive
Chicago, IL 60604-1229
United States of America

State of Nationality

State of Residence

Telephone No.
312 201 2000

Facsimile No.
312 201 2555

Teleprinter No.

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

☒ the receiving Office
☐ the International Searching Authority
☒ the International Preliminary Examining Authority
☐ the designated Offices concerned
☒ the elected Offices concerned
☐ other:

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer
Sean Taylor

Telephone No.: (41-22) 338.83.38

Form PCT/IB/306 (March 1994)

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